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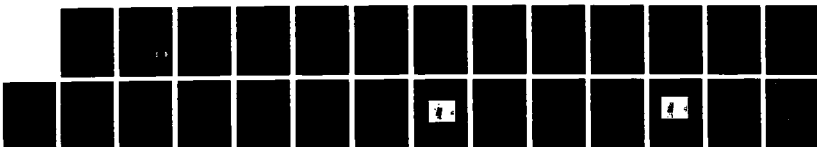
REACTIVE PLASTICIZERS IN NEGATIVE E-BEAM RESISTS(U)  
CORNELL UNIV ITHACA SCHOOL OF CHEMICAL ENGINEERING  
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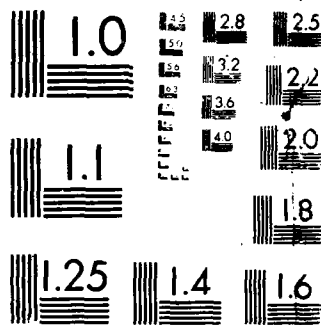
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Technical Report No. 7

## REACTIVE PLASTICIZERS IN NEGATIVE E-BEAM RESISTS

by

Y. M. N. Namaste, S. K. Obendorf, and F. Rodriguez

Prepared for presentation at the  
32nd International Symposium on  
Electron, Ion, and Photon Beams  
May 31-June 3, 1988  
Fort Lauderdale, FL

Olin Hall, Cornell University  
School of Chemical Engineering  
Ithaca, NY 14853

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## 19. Abstract (Cont'd)

Blends of the reactive monomers with fractionated base polymers with low molecular weight and low polydispersities are promising. Low molecular weight PCMS and VMCH exhibit good contrast and resolution, but suffer from poor sensitivity. Addition of reactive monomers to these base polymers increases their sensitivity, while maintaining contrast and resolution that is superior to that of the high molecular weight unfractionated polymer alone. These blends exhibit less swelling than the host polymers.

The mechanism is believed to be that the monomer, which plasticizes the host polymer, polymerizes and crosslinks upon exposure, resulting in an interpenetrating network (IPN). The plasticized polymer in the unexposed resist film readily dissolves in the developing solvent, whereas the glassy IPN in the exposed region has limited solubility.

# Reactive Plasticizers in Negative E-Beam Resists

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## ABSTRACT

Negative electron resists generally exhibit excellent sensitivity, but suffer from swelling during development which results in poor resolution. A new approach to this problem is presented, in which reactive monomers are blended with host polymers to provide sensitive negative resists with improved resolution. Polychloromethylstyrene (PCMS) and VMCH (a terpolymer containing 86% vinyl chloride, 13% vinyl acetate and 1% maleic acid) were both found to be compatible with two reactive monomers, trimethylolpropanetrimethacrylate (TMPTMA) and dipentaerythritolpentaacrylate (DPEPA). Addition of 20% (w/w) of either monomer to PCMS or VMCH resulted in approximately ten fold increases in sensitivity. Sensitivity increases dramatically with increasing monomer content, with a 1:1 blend of VMCH and DPEPA exhibiting a sensitivity of about  $0.1 \mu\text{C}/\text{cm}^2$ . This represents more than a 100-fold increase in sensitivity over that of VMCH alone. These blends exhibit resolution of  $0.5 \mu\text{m}$ .

Blends of the reactive monomers with fractionated base polymers with low molecular weight and low polydispersities are promising. Low molecular weight PCMS and VMCH exhibit good contrast and resolution,



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#### INTRODUCTION

Negative-working electron resists, such as polychloromethylstyrene (PCMS), are available with excellent sensitivity in the 0.1 to 1  $\mu\text{C}/\text{cm}^2$  range.<sup>1</sup> However, the exposed, crosslinked polymers swell during development, often leading to bridging and oscillatory distortion (snaking). These factors, as well as poor contrast, severely limit the resolution of most of the currently available negative electron resists.<sup>2,3</sup> Researchers have pursued various approaches to minimize the problems associated with swelling, and improve the resolution, of negative resists, including: 1) increasing the glass transition temperature of the resist<sup>4</sup>, 2) optimizing the developing system,<sup>5,6</sup> 3) narrowing the molecular weight distribution,<sup>1,7,8</sup> 4) employing image reversal techniques with positive resists,<sup>9</sup> and 5) producing resists that change solubility by

means other than crosslinking.<sup>10</sup>

In the current work, we have investigated the use of reactive monomers in resists as a means of providing negative resists with improved resolution and sensitivity. Purely monomeric resists would be interesting in that they could polymerize upon irradiation, resulting in a dramatic difference between exposed and unexposed material. Unfortunately, monomers do not possess the film-forming properties of polymers, and thus would be difficult to use alone as resists. We have therefore investigated blends of reactive monomers with host polymers. Provided that the monomer/polymer pairs are adequately miscible, the blends form homogeneous films of plasticized polymer prior to exposure. Upon exposure, the monomer polymerizes and crosslinks, reducing the solubility of the exposed material.

There is an interesting similarity of this work to that of Taylor and co-workers, who blended resists with metal-containing reactive monomers.<sup>11,12</sup> In these systems, the unreacted monomer in unexposed regions is evaporated during a post-exposure bake step, resulting in a plasma-developable image. Wet development of these blends has apparently not been reported.

Blends of polymers and reactive monomers have also been used in the coatings industry. For some years, inks and coatings have been formulated with combinations of polymers and reactive solvents as a means of satisfying air-pollution control regulations.<sup>13</sup> The idea is to replace the volatile solvent in a conventional lacquer by a polymerizable monomer. The polymer-monomer solution is converted to a solid film by polymerization of the monomer, usually induced by



ultraviolet light or heat. Another technological area with some relevance to the present work is that of peroxide (thermal) crosslinking of polyolefins. It is known that addition of small amounts of polyfunctional vinyl monomers increase the crosslinking yield. As an example, a few percent of triallyl isocyanurate can be added to a low density polyethylene containing a crosslinking agent like dicumyl peroxide in order to make a cable insulation material.<sup>14</sup>

The base polymers studied in the current work include PCMS and VMCH (a terpolymer consisting of 86% vinyl chloride, 13% vinyl acetate and 1% maleic acid). The most promising monomers investigated were trimethylolpropanetrimethacrylate (TMPTMA) and dipentaerythritol-pentaacrylate (DPEPA). We have evaluated the suitability of blends of these polymer/monomer combinations for lithography in terms of sensitivity, contrast and resolution. We also have studied the effects on lithographic performance of blend composition, host polymer fractionation, prebake conditions, developing system and post-exposure vacuum residence time.

## EXPERIMENTAL

### Materials:

High molecular weight PCMS was synthesized by free-radical polymerization (in bulk) of chloromethylstyrene at 50°C, initiated with 2,2'-azobisdimethylvaleronitrile (ADVN). Low molecular weight PCMS was synthesized in a 10% solution in methyl ethyl ketone at 80°C, using 2,2'-azobisisobutyronitrile (ABBN) as the initiator. VMCH (a commercial terpolymer consisting of 86% vinyl chloride, 13% vinyl acetate, and 1%

maleic acid) was obtained from Union Carbide and was fractionated from tetrahydrofuran by precipitation with methanol. Molecular weights of the host polymers were determined by gel permeation chromatography (GPC), relative to polystyrene standards.

Dipentaerythritol pentaacrylate (DPEPA) was obtained from Monomer, Polymer and Dajac Laboratory, Inc. Trimethylolpropanetrimethacrylate (TMPTMA) was obtained from Rohm and Haas Co. TMPTMA was supplied with 200 ppm of monomethyl ether of hydroquinone (MEMQ) to inhibit spontaneous polymerization. Removal of inhibitor from this monomer had little effect on the lithographic properties of the blends. Compatibility of the reactive monomers with the host polymers was determined by dissolving them in a mutual solvent (chlorobenzene), and spin-casting films of the blends. The films were baked at various temperatures and subjected to high vacuum, and then observed with light microscopy (1000x) for evidence of phase separation.

#### Lithographic evaluation

The blends were spin-coated onto silicon wafers from solutions in chlorobenzene with spin speeds around 2000 RPM for 60 seconds. The films were not prebaked prior to exposure except as indicated. Lithographic test patterns were exposed with a Cambridge Instruments EBMF 10.5 at the National Nanofabrication Facility at Cornell University. The accelerating voltage was 20 kV, and no proximity correction was used.

All patterns were developed in methyl isobutyl ketone (MIBK) for 30 seconds while spinning at 500 RPM, followed by a 20 second rinse in

2:1 methyl ethyl Ketone (MEK): isopropyl alcohol (IPA), also at 500 RPM. Film thicknesses were measured with a Leitz interferometer. Pattern resolution was evaluated using a Cambridge Instruments SEM.

## RESULTS

Compatibility of the reactive monomers with the host polymers is essential. Two monomers that exhibited excellent compatibility with both PCMS and VMCH were trimethylolpropanetrimethacrylate (TMPTMA) and dipentaerythritol pentaacrylate (DPEPA). The chemical structures of these monomers are shown in Figure 1. Blends with these monomers showed no signs of microphase separation under any of the process conditions encountered. Several of the other monomers investigated did show severe problems with phase separation. These included polyethylene glycol (600) dimethacrylate, polypropylene glycol (2000) dimethacrylate, and tributyl tin acrylate.

### Sensitivity:

Both TMPTMA and DPEPA were found to significantly enhance the sensitivity of the PCMS and VMCH host polymers. Addition of 20% (w/w) of either of these monomers resulted in five to twenty-fold increases in sensitivity (Table I). Blends with greater than 20% TMPTMA were not evaluated because TMPTMA was found to be volatile under high vacuum from blends containing more than 20% TMPTMA. In contrast, the DPEPA monomer was completely non-volatile under the conditions of lithographic evaluation. Increasing the DPEPA concentration in blends with VMCH dramatically increased sensitivity, with a 1:1 blend of VMCH and DPEPA exhibiting a sensitivity of about  $0.1 \mu\text{C}/\text{cm}^2$  (Table II).

This represents more than a 100-fold increase in sensitivity over that of VMCH alone.

#### Contrast and Resolution:

Coinciding with the improved sensitivity of these blends, there is a reduction of contrast, relative to the host polymers. For example, the addition of 20% TMPTMA to PCMS reduces the contrast ( $\gamma$ ) of this resist from about 1.2 to 0.9 (Figure 2). Contrast of the blends is improved somewhat by fractionation of the host polymer, as discussed below.

Resolution of 0.5  $\mu\text{m}$  is achieved with PCMS or VMCH with 20% of either TMPTMA or DPEPA. Patterns with 0.5  $\mu\text{m}$  features exposed at 2  $\mu\text{C}/\text{cm}^2$  in 4:1 VMCH:TMPTMA are shown in Figure 3.

#### Processing Conditions:

Most of the exposures used for lithographic evaluation of these blends were conducted using films of the blends that were not prebaked prior to exposure. Blends containing TMPTMA were found to form insoluble residues after prebaking for one hour above 80°C. Similarly, the DPEPA blends exhibit limited solubility after prebaking at temperatures above 100°C. Using a prebake temperature of 90°C, blends with DPEPA show little effect of prebake on lithographic response (Figure 4).

The blends exhibit symptoms of post-exposure reaction if left in the vacuum system of the exposure tool after exposure. This so-called "dark reaction" serves to increase the apparent sensitivity of the blends with increasing storage time in the vacuum (Figure 5). This post-exposure reaction is well-documented in the literature for other

negative electron resists.<sup>15</sup> It has been shown that addition of a radical scavenger to the resist can reduce this reaction and improve contrast,<sup>15</sup> but this has not been attempted here.

#### Fractionation and Low Molecular Weight Host Polymers:

It is well known that reduction of the molecular weight of a crosslinking resist reduces sensitivity while improving contrast and resolution.<sup>1</sup> Reduction of the molecular weight distribution further improves the contrast of negative resists.<sup>1,7,8</sup>

Blends of the reactive monomers with fractionated VMCH and with low molecular weight PCMS are promising (Figure 6). The low molecular weight host polymers exhibit poor sensitivity, but high contrast. Addition of reactive monomer to these polymers increases their sensitivity, yielding sensitivities similar to that of the blends with the higher molecular weight host polymers. Some of the contrast of the low molecular weight material is maintained, resulting in both higher sensitivity and higher contrast for the blend than for high molecular weight polymer alone (Figure 6).

Fractionation of the host polymer also greatly improves the resolution of the blends. Use of low molecular weight polymer reduces the scumming around exposed patterns, resulting in better resolution. For example, fractionated VMCH ( $M_n = 30,100$ ;  $M_w = 42,000$ ;  $PD = 1.4$ ) with 20% DPEPA provides very clean  $0.5 \mu m$  patterns using an exposure dose of  $2 \mu C/cm^2$  (Figure 7). Similar results are obtained for blends with low molecular weight PCMS.

## DISCUSSION

The observed stability of the exposed patterns is believed to be due to the conversion of the polymer-plasticizer system from a rubbery state (with high crosslinking yield) to a glassy interpenetrating network (IPN), resulting in a low diffusivity of the developing solvent in the exposed regions. The impaired mobility of the polymer chains in an IPN stabilizes the system, and prevents separation into microphases at higher temperatures. Meanwhile, the unexposed film remains very soluble due to plasticization by the monomer. There is also a high probability that the base polymer becomes covalently linked to the polymerized plasticizer during irradiation.

Future work with this resist system should be very promising. Optimization of these resists in terms of blend composition and exposure conditions is expected to further improve resolution, contrast, sensitivity and film stability. In addition, these blended systems have potential for use in X-ray and deep UV lithography. Preliminary experiments further indicate that the incorporation of organometallic compounds into these blends results in enhancement of both oxygen RIE etch resistance and sensitivity, and may lead to a resist system with dry-developing capability.

## ACKNOWLEDGEMENTS

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TABLE I

Effect on Sensitivity of Addition of 20% (w/w) Reactive Monomer  
to Host Polymers

| Host Polymer      | Sensitivity, $D_{g0.5}$ ( $\mu\text{C}/\text{cm}^2$ ) |             |              |
|-------------------|---|-------------|--------------|
|                   | Polymer Only  | w/20% DPEPA | w/20% TMPTMA |
| VMCH <sup>1</sup> | 23  | 0.90        | 4.0          |
| PCMS <sup>2</sup> | 1.8   | 0.35        | 0.25         |

<sup>1</sup>VMCH not fractionated, as received from manufacturer:  
 $M_n = 47,600$ ;  $M_w = 85,400$ ; PD = 1.8

<sup>2</sup>PCMS not fractionated, as synthesized:  
 $M_n = 77,200$ ,  $M_w = 145,800$ ; PD = 1.9

TABLE II

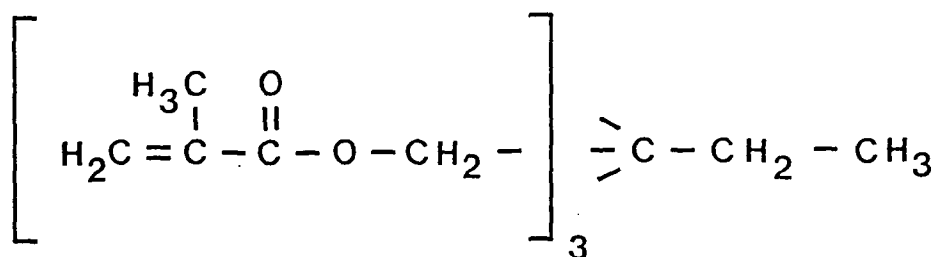
Sensitivity of VMCH/DPEPA Blends with Varying Blend Compositions

| <u>DPEPA Concentration</u><br>(% w/w) | <u>50% Gel Dose, <math>D_{g0.5}</math></u><br>( $\mu\text{C}/\text{cm}^2$ ) |
|---------------------------------------|---|
| 0                                     | 23.0  |
| 10                                    | 2.6   |
| 20                                    | 0.9   |
| 33                                    | 0.6   |
| 50                                    | 0.15  |

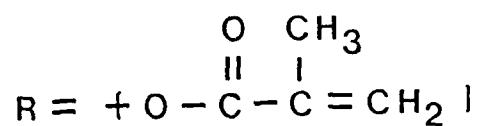
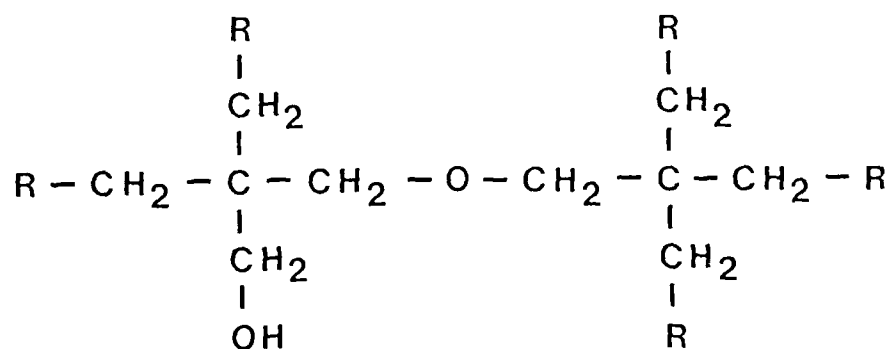
#### FIGURE CAPTIONS

- Figure 1: Chemical structures of the reactive monomers TMPTMA and DPEPA.
- Figure 2: Lithographic response of a 4:1 blend of PCMS and TMPTMA.
- Figure 3: 0.75 and 0.50  $\mu\text{m}$  patterns exposed in 4:1 VMCH:TMPTMA at 2  $\mu\text{C}/\text{cm}^2$ . The patterns were developed for 30 seconds in MIBK, followed by a 20 second rinse in 2:1 MEK:IPA.
- Figure 4: Effect of prebaking on the lithographic response of 4:1 VMCH:DPEPA.
- Figure 5: Effect of vacuum curing on the lithographic response of 4:1 VMCH:DPEPA.
- Figure 6: Effects of fractionation of VMCH on contrast and sensitivity of the host polymer and a 4:1 blend of VMCH and DPEPA.
- Figure 7: Resolution of fractionated VMCH ( $M_n=30,100$ ;  $M_w=42,000$ ;  $PD=1.4$ ) with 20% DPEPA. This pattern was exposed with a dose of 2  $\mu\text{C}/\text{cm}^2$  at 20kV. Feature sizes are 0.75 and 0.50  $\mu\text{m}$ , as indicated.

Figure 1



Trimethylolpropanetrimethacrylate (TMPTMA)



Dipentaerythritol pentaacrylate (DPEPA)

Figure 2

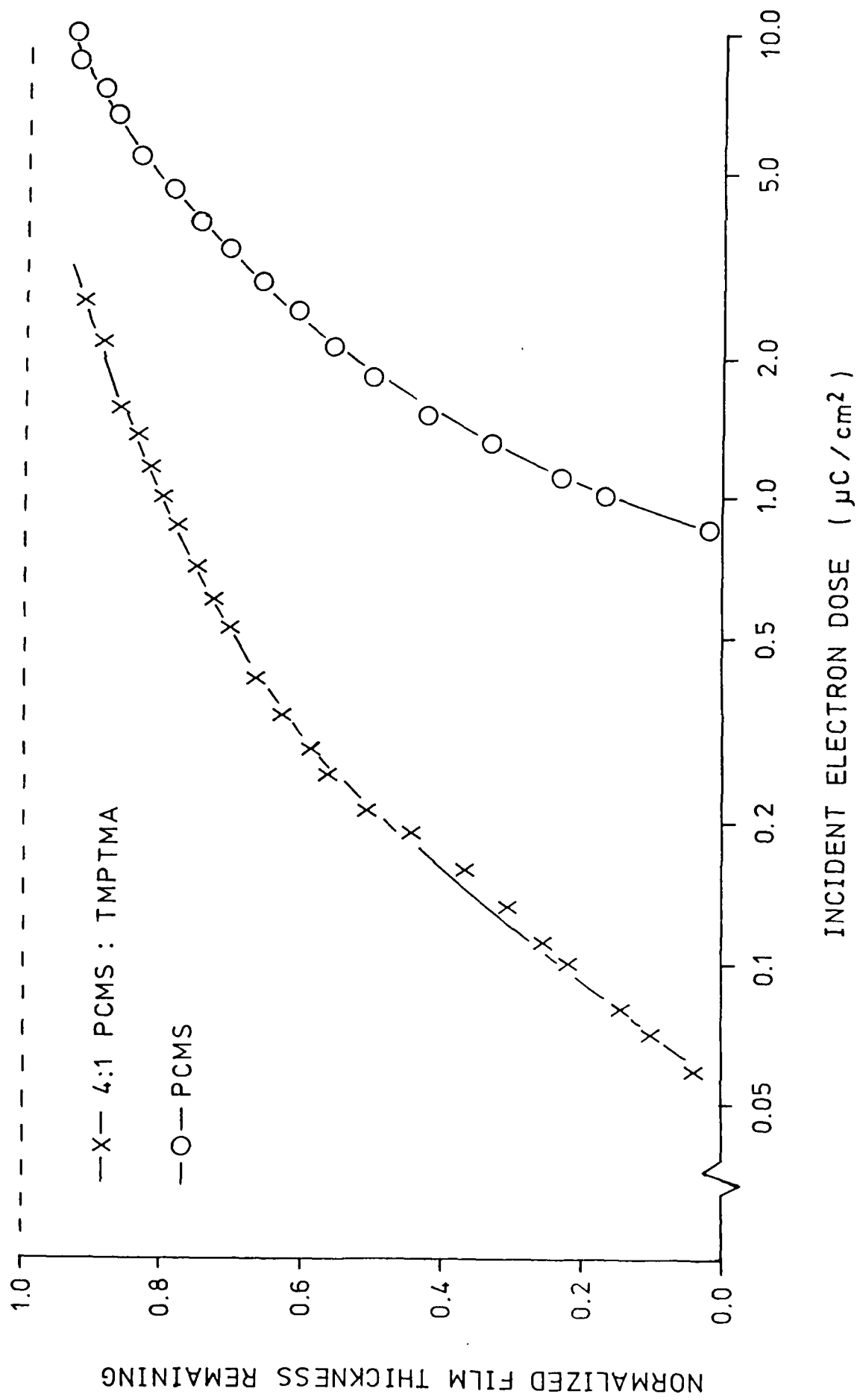


Figure 3

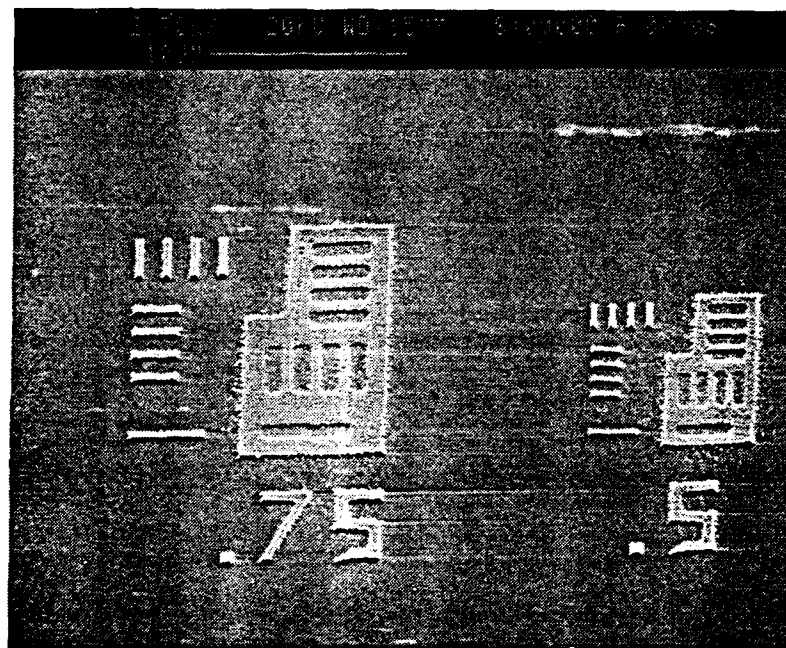


Figure 4

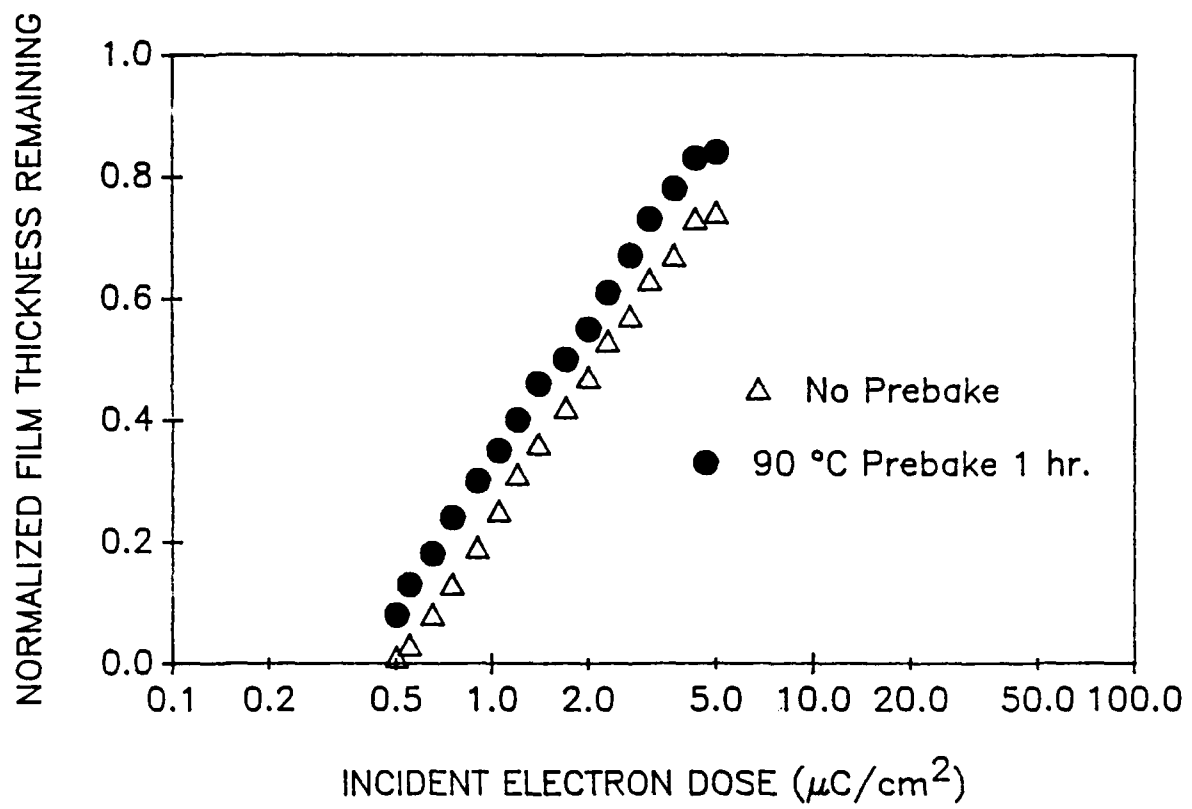


Figure 5

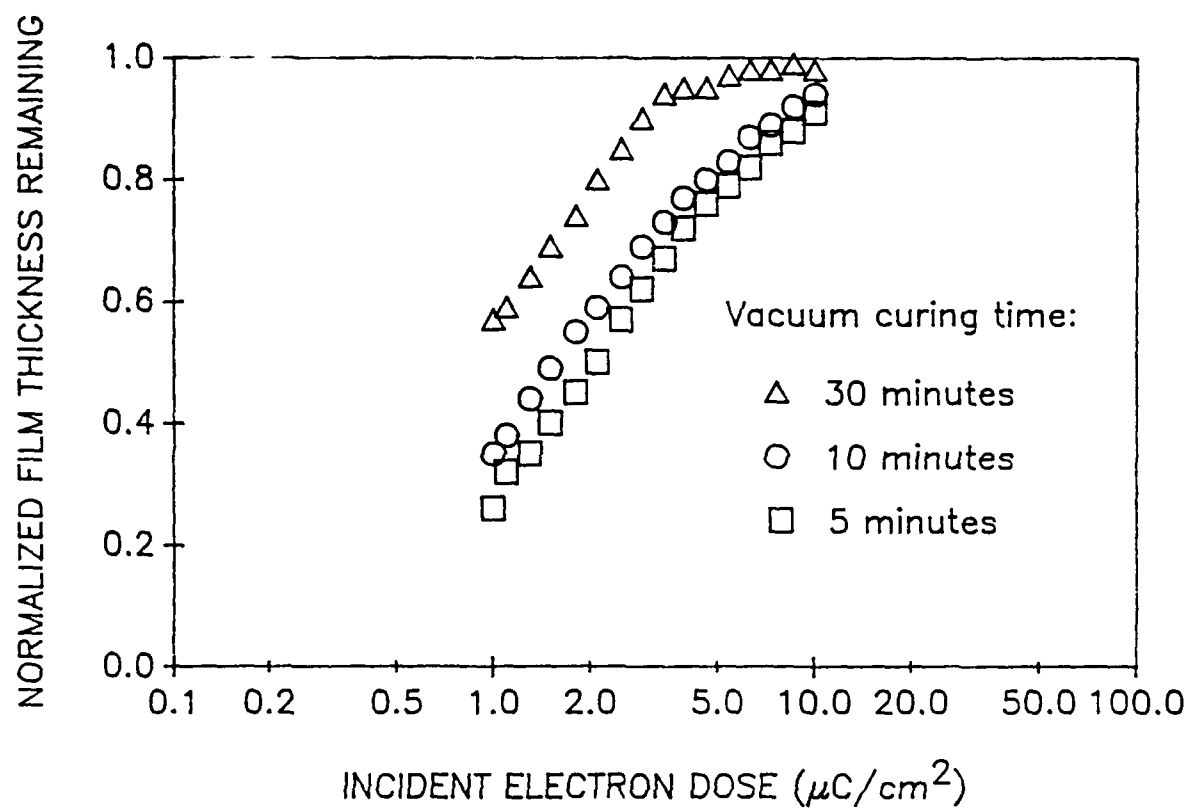




Figure 6

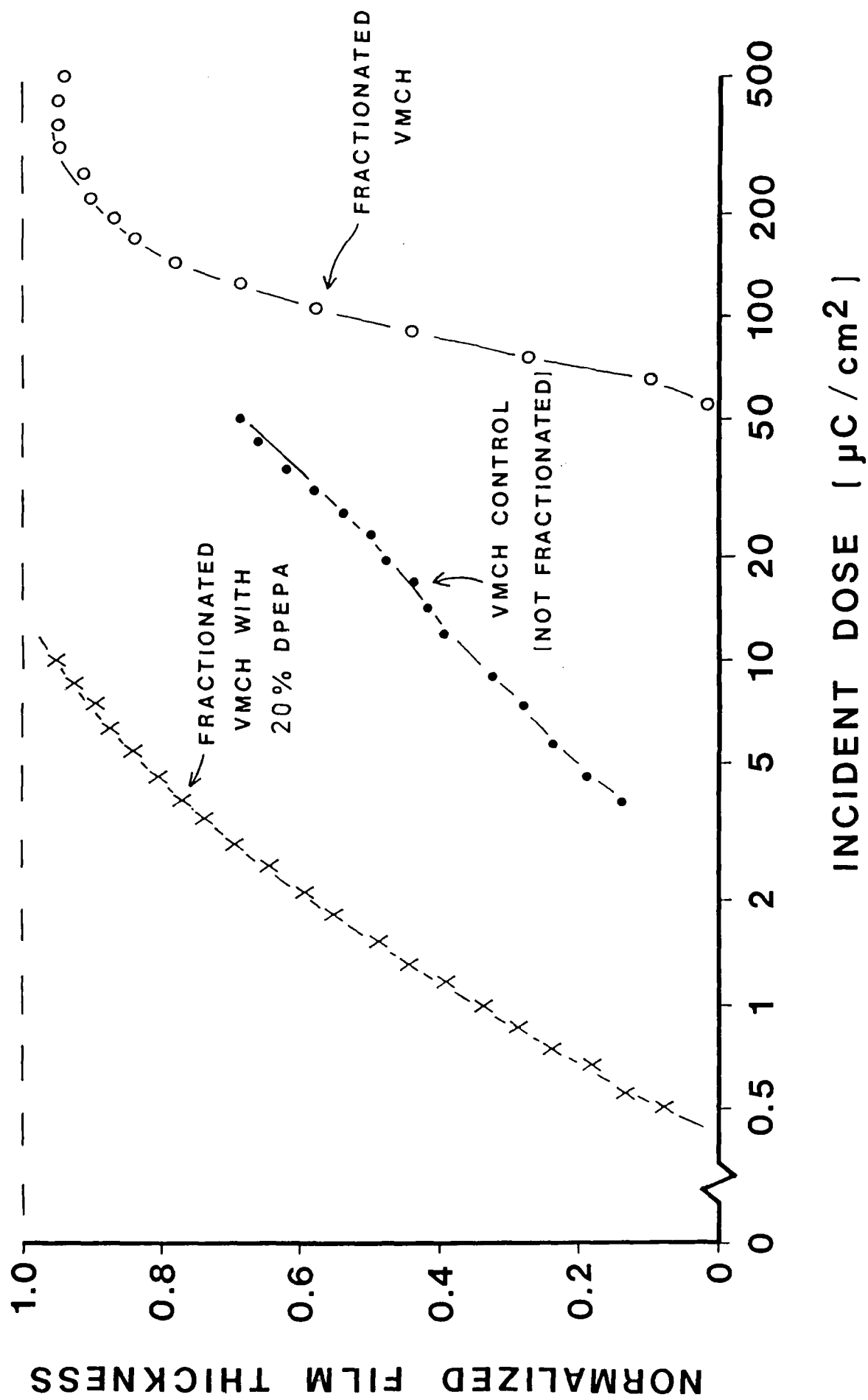
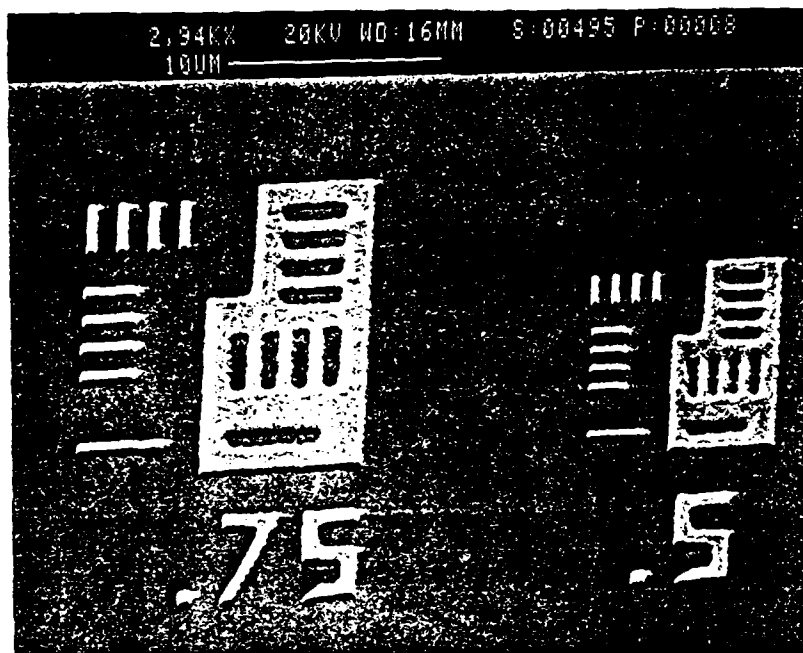


Figure 7



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